(54) Title: IMPROVEMENTS IN AND RELATING TO MATERIAL TESTING

(57) Abstract: Methods of corrosion testing metals are provided, including a method which provides a sample of the metal, a metal including grains, contacting the sample of the metal with a test liquid, the rate of corrosion of the sample of metal in the test liquid being measured, wherein the sample of the metal is contacted with a treatment liquid prior to contact with a test liquid, the contact with the treatment liquid giving grain dropping from the metal. In this way accurate corrosion testing is achieved in far less time than using prior art techniques. The invention also includes methods for establishing the rate of corrosion for a particular surface of a multi-surfaced metal.

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# IMPROVEMENTS IN AND RELATING TO MATERIAL TESTING

This invention concerns improvements in and relating to material testing, particularly to corrosion testing of steel specimens exposed to corrosive liquids.

Corrosion is a factor which needs to be accounted for in a wide range of situations within the nuclear, chemical, offshore oil and other industries. It is desirable to be able to predict corrosion as accurately as possible.

Some materials, particularly stainless steels, are known to demonstrate an apparent corrosion rate which initially increases with time upon exposure to corrosive solutions. Eventually a constant corrosion rate is reached, but the level of this constant corrosion rate and the time at which it onsets cannot be found without specific testing for protracted periods of time. Determining this rate is an important issue.

The present invention has amongst its aims the provision of corrosion testing which is accurate but requires less time to perform. The present invention has amongst its aims the provision of a pre-corrosion treatment for use in corrosion testing.

Corrosion of materials is also known to be anisotropic in many cases, with the level of variation between surfaces formed in different ways, such as plate compared with cut surfaces, being unknown.

The present invention also has amongst its aims to determine interrelationships between the corrosion rates for different surfaces of a material.

According to a first aspect of the invention we provide a method of corrosion testing metals, the method including providing a sample of the metal, the metal including grains, and contacting the sample of the metal with a test liquid, the rate of corrosion of the sample of metal in the test liquid being measured, wherein the sample of the metal is contacted with a treatment liquid prior to contact with the test liquid, the contact with the treatment liquid giving grain dropping from the metal.

The metal may be elemental, but is preferably an alloy. The metal may be stainless steel, for instance austenitic stainless steel and/or steel stabilised by Nb.

The sample may be rectilinear. Preferably the sample has a first face, and ideally a pair of first faces, formed by contact with the rollers during production. Preferably the

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sample has a second face, and ideally a pair of second faces, formed perpendicular to the rolling direction during production. Preferably the sample has a third face, and ideally a pair of third faces, formed perpendicular to both the first and second faces.

The grains are preferably part of the microstructure of the metal.

The sample may be contacted with the treatment liquid by immersion within it. The sample may be contacted with the treatment liquid by hanging in it.

The treatment liquid may include nitric acid, particularly of 5M or greater, most preferably 8M or greater. The treatment liquid may include one or more oxidants. Preferably at least 0.5g/l Cr(IV) is provided in the treatment liquid and ideally at least 1.0g/l Cr(IV) is provided. The Cr(IV) may be added as $CrO_3$.

The sample may be provided in contact with the treatment liquid for at least 12 hours, more preferably at least 18 hours and ideally 24 hours. It is particularly preferred that the sample is in contact with the treatment liquid until grain dropping is observed. Grain dropping may be observed by visual and/or optical inspection of the liquid below the sample, for instance for the presence of grains below the sample in the treatment liquid, and/or by a measured reduction in the size of the sample or one or more surfaces thereof and/or by visual and/or optical examination of the sample. One or more treatments with the treatment liquid may be provided.

The treatment liquid may be provided at a temperature of at least 80°C, more preferably at least 90°C, still more preferably at least 100°C, even more preferably at least 110°C and ideally boiling.

The treated sample may be contacted with the test liquid by immersion within it. The sample may be contacted with the test liquid by hanging in it.

Preferably the test liquid will cause intergranular corrosion of the sample of metal.

The test liquid may include nitric acid, particularly aqueous nitric acid solutions. The nitric acid may contain one or more dissolved species, such as oxidants. The oxidants may be or include one or more of $Ce^{4+}$, $CrO_4^{2-}$, $Cr_2O_7^{2-}$, $Fe^{3+}$, $NO_3^-$, iron or ferric nitrate.

The test liquid may be provided at a temperature of at least 80°C, more preferably at least 90°C and ideally 100°C. The test liquid may be provided at a thermostatically controlled temperature.

The rate of corrosion may be measured by periodic weighing of the sample. The sample may be washed with water and/or methanol prior to weighing. The sample may be

dried prior to weighing. The mass of sample lost may be converted into a corrosion rate expressed in terms of a thickness of loss from the sample. The thickness loss may be obtained by evenly attributing the metal loss to the surface area of the remaining sample.

According to a second aspect of the invention we provide a method of corrosion testing metals, the method including providing a sample of the metal, the metal including grains, and contacting the sample of the metal with a test liquid, the rate of corrosion of the sample of metal in the test liquid being measured, wherein the sample of the metal is contacted with a treatment liquid prior to contact with the test liquid, the contact with the treatment liquid being for at least 6 hours and the treatment liquid including at least 5M acid and one or more oxidants.

The second aspect of the invention may include any of the features, options or possibilities set out elsewhere in this document, including those of the first aspect in particular.

According to a third aspect of the invention we provide a method of establishing the rate of corrosion for a first surface of multi-surfaced metal in contact with a test liquid, the method including

contacting a first test sample of the metal with the test liquid, the first test sample having a first surface which experiences a constant corrosion rate on initial contact with the test liquid and other surfaces which undergo a non-constant corrosion rate, or non-constant corrosion rates, on initial contact with the test liquid and

contacting a second test sample of the metal with the test liquid, the second test sample being of equivalent form to the first test sample, the second test sample having all surfaces which undergo a non-constant corrosion rate, or non-constant corrosion rates, on initial contact with the test liquid,

the first and second test samples being contacted with the test liquid for the same time period, the differences in metal loss between the first sample and the second sample being attributed to the first surface of the first sample.

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The first surface may be an external surface formed during production of the metal, particularly the surface contacted by the rollers, the plate surface. The first surface may be a cut surface, for instance one parallel to the rolling direction and perpendicular to the plate surface, the side surface. The first surface may be a cut surface, for instance one perpendicular to the rolling direction and perpendicular to the side surface, the end surface. The end surface generally undergoes corrosion faster than the side surface and both generally undergo corrosion faster than the plate surface. The surfaces of the sample are preferably perpendicular to one another. Preferably pairs of opposing surfaces are provided.

The first surface may experience constant rate corrosion on initial contact due to previous contact with a treatment liquid. The treatment liquid is preferably provided according to the first aspect of the invention.

The other surfaces may experience non-constant corrosion on initial contact due to the surfaces not having contacted the treatment liquid, or more preferably due to the removal from that surface of the part of the surface which has been corroded by contact with the treatment liquid. The part of the surface may be removed by cutting and/or grinding.

The rate of corrosion of the first surface may be obtained by attributing the metal loss of the second sample evenly to all surfaces to provide a metal loss per unit area value, for instance expressed per $cm^2$, and applying the same metal loss per unit area to the non-first surfaces of the first sample to give a non-first surface metal loss, the remaining metal loss from the first sample being attributed to the first surface of the first sample.

The differences may be expressed in terms of a mass, but may be converted into a thickness for that surface or the whole sample.

The metal loss attributed to the first surface may be corrected to reflect the metal lost from the first surface of the second sample due to non-constant corrosion on all the surfaces except that corresponding to the first surface of that sample.

The method may be applied to each of the situations where the plate, side and end surface is the first surface. The results for the three surface types may be used to calculate an overall thickness loss or corrosion rate for the sample. This may be compared with the result from a test sample in which all surfaces are provided so as to corrode at a constant rate from initial contact with the test liquid.

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A ratio of the corrosion rate of the plate surface to the end surface and/or the side surface may be obtained. A ratio of the corrosion rate of the end surface to the plate surface and/or the side surface may be obtained. A ratio of the corrosion rate of the side surface to the end surface and/or the plate surface may be obtained. Preferably a ratio of the rate of corrosion of the plate surface to the rate of corrosion of the overall sample is obtained.

The results may be verified by conducting the method at different temperatures and/or different strengths of acid and/or different dissolved species and/or different oxidants in the test liquid.

The third aspect of the invention may include any of the features, options or possibilities set out elsewhere in this document.


Various embodiments of the invention will now be described, by way of example only, and with reference to the accompanying drawings, in which:-

Figure 1 is a scanning electron micrograph showing light etching levels of corrosion at grain boundaries for a sample of stainless steel;

Figure 2 is a scanning electron micrograph showing intense penetration and undercutting of grains for a stainless steel sample;

Figure 3 illustrates different penetration of corrosion into a plate surface, cut surface parallel to rolling and a cut surface perpendicular to rolling;

Figure 4 illustrates the variation in corrosion rate with time for an 18/13/1 stainless steel sample in 8M nitric acid containing 25g\l iron at a solution temperature of 100°;

Figure 5 illustrates corrosion rates against time for pre-corrosion treated and standard test samples exposed to nitric acid;

Figure 6 illustrates the design of various test specimens used in the investigations behind the present invention; and

Figure 7 illustrates the production of plate, side and end surfaces during the production of metal sheet.


The prediction of material corrosion is desirable to ensure safe plant design and effective maintenance and replacement programs for plant during their operation.

Corrosion is a particular issue where materials are exposed to corrosive solutions as frequently happens in the nuclear, chemical, offshore oil and other industries. Even where corrosion resistant materials are used, some corrosion may occur and has to be accounted for.

Stainless steels generally exhibit good passivity in many corrosive solutions including nitric acid solutions. However, in sufficiently oxidising conditions stainless steels can suffer inter-granular attack and the corrosion rate can become appreciable as a result. Such oxidising conditions include, but are not limited to, strong aqueous solutions of nitric acid at or close to boiling point, or weaker solutions of nitric acid which incorporate powerful oxidants such as $Ce^{4+}$, $CrO_4^{2-}$ or $Cr_2O_7^{2-}$. Inter-granular attack occurs owing to the fact that grain boundaries dissolve much faster than the metal matrix. The intensity of such attack depends on the oxidising power of the solution and may range from light etching of the grain boundaries as illustrated in Figure 1 to intense penetration which will eventually undercut the grain and lead to it falling away from the surface as illustrated in Figure 2.

Significantly, micro-structural effects arising from the steel making process can give rise to strong anisotropic corrosion of stainless steel samples in nitric acid solutions, depending on the orientation of the exposed grains at a surface. The most pronounced appearance of this is end-grain corrosion generally occurs in the end surface, that is preferential attack parallel to the hot-working direction of the sample. Other surfaces, the plate surface formed by the rolls in production and the side surface, generally suffer lower corrosion penetration with time. Figure 3 illustrates corrosion penetration for the plate surface (upper illustration), a cut surface parallel to the rolling direction (middle illustration) and the cut surface perpendicular to the rolling direction (lower illustration). As can be seen far higher rates of penetration occur in the latter case. Prediction of corrosion rates for the whole of the sample and for individual surfaces of a sample is desirable to enable effective plant design and maintenance. Corrosion prediction from test samples is particularly difficult as cut surfaces on samples are frequently exposed to the corroding solution during test work, but are not usually exposed to the process on plant.

A variety of investigative techniques have been employed to effect corrosion testing but none offers sufficiently reliable and quick corrosion testing to be ideal. The most commonly used form is immersion testing of coupons/samples containing cut

surfaces. Such coupons, however, have exposed end and side grain surfaces and accordingly run the risk of generating unrealistically high corrosion rates when compared with plant conditions. An additional problem with some materials, such as stainless steel, is that the corrosion rate appears to increase with immersion time until a steady state corrosion rate is reached. This general principle is illustrated in Figure 4. Significantly the immersion time required to reach the constant corrosion rate depends on how corrosive the solution is and the nature of the sample being tested. This time period can be very long (several thousand hours), but unless the testing is continued for sufficiently long to reveal the steady state rate, it is possible that the rate of plant corrosion could be significantly underestimated.

The present invention, detailed below, includes within in a method for shortening the time required to determine the long-term corrosion rate (steady state corrosion rate).

In basic terms the method for shortening the test time involves a pre-corrosion treatment of the test sample which converts it in a relatively short space of time into a sample which will subsequently undergo steady state corrosion under the desired test conditions, with that steady state corrosion rate matching the steady state corrosion rate which would occur merely through far more prolonged exposure to the test liquid.

The effectiveness of the technique and details of it are discussed below in the context of the specific experimental results.

The present invention also provides details of the relationship between corrosive losses from different surface types that allow accurate corrosion testing for real life situations where only plate surfaces are exposed, even though all surfaces are exposed to the corrosive liquid in the sample testing.

## Preparation of Test Samples

The specimens used to demonstrate the effectiveness of the technique were manufactured from 18/13/1 stainless steel, which is an Nb stabilised austenitic steel used in the construction of some plants of the applicant and of other organisations. Analysis of such steel gives the chemical composition (weight %) set out in Table 1 below.

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Table 1

| Element | Weight % |
|---------|----------|
| Fe | 67.65 |
| C | 0.09 |
| Si | 0.51 |
| Mn | 0.99 |
| Cr | 16.6 |
| Ni | 12.6 |
| Mo | 0.08 |
| Cu | 0.13 |
| Ti | 0.02 |
| Nb | 0.92 |
| Al | 0.02 |
| V | 0.10 |
| W | 0.05 |
| Co | 0.19 |
| S | 0.006 |
| P | 0.07 |

The plate used to form the test samples was solution annealed at 1050°C for one hour followed by quenching so as to ensure a reproducible microstructure.

Each sample, which was nominally of dimensions 15x10x4mm was provided with a 1.5mm diameter hole to enable it to be suspended from a hook made from NAG (nitric acid grade) 19Cr/9Ni low-carbon welding wire during testing and, where applicable, pre-treatment. The specimens were all wet-ground with SiC paper to 800 grit. The test samples prepared in this way were then either corroded or pre-corrosion treated and then corroded in the manner set out below.

**Pre-corrosion Treatment**

The pre-corrosion treatment involved placing the sample in boiling 8M nitric acid containing 1g/l Cr(VI) added as $CrO_3$ for 24 hours. This is a convenient treatment as it is

inherently self-limiting due to the reduction of Cr(VI) to Cr(III). The pre-corrosion treatment is sufficiently aggressive to initiate wholesale grain dropping on 18/13/1 stainless steel. Repeated treatments can be used for higher grades of nitric acid resistant materials.

Following the pre-corrosion treatment the samples were washed in methanol and then weighed to 0.1mg accuracy prior to immersion in the corrosion test liquor.

## Corrosion Treatment

The corrosion test liquor itself was of either 8M or 9M $HNO_3$ containing 15g/l Fe (added as $Fe(NO_3)_3.9H_2O$), 12.5g/l Ru (added as $RuNO(NO_3)_3$ solution), 8.48g/l Pd (added as $Pd(NO_3)_2$ solution), and 172.5g/l $NO_3^-$ (added as 118.3g/l $NaNO_3$ and 140.5g/l $KNO_3$). The solutions were chosen to simulate process liquors of interest to the applicant, but other process liquors were applicable to other situations could be investigated in a similar way. The corrosion test liquor was maintained at 100°C using a thermostatically controlled bath filled with undiluted glycol.

The corrosion rate of the specimens was measured periodically throughout the test by removing the specimens from the solution at 168 hour intervals, washing the sample with de-ionised water and then methanol. No ultrasonic or mechanical cleaning of the sample was undertaken so as to avoid the dislodgement of grains and hence weight loss. After being dried the test samples were weighed and a corrosion rate for that period was calculated from the weight loss, and the nominal surface area of the whole specimen. After this the specimens were replaced in the test solution for the continuing test. #

## Test Results – Comparison of Corrosion of Pre-Corrosion Treated and Untreated Samples

The period corrosion rate of initially ground 18/13/1 stainless steel specimens of the above mentioned type, as a function of a motion type, is illustrated in Figure 5 by the solid black lines. Two samples using 8M nitric acid and two samples using 9M nitric acid were considered.

The Figure 5 plot also shows the corrosion rates with time obtained for pre-corrosion treated samples of the same material; dotted black lines. The data for the pre-corrosion treated specimens is plotted with the time offset to allow direct comparison with

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the behaviour of the samples that were not treated before being subjected to the corrosion test.

As can be seen the two ground samples which were not subjected to the pre-corrosion treatment undergo the type of increasing corrosion rate with time illustrated in Figure 4. In the latter part of the test time periods the steady state corrosion has been reached. Extrapolation of the data sets reveals a transition point between increasing corrosion rate and steady state corrosion rate at approximately 810 hours for the 9M nitric acid solution and at approximately 900 hours for the 8M nitric acid solution.

Significantly, in the case of the pre-corrosion treated samples, the periodic corrosion rate was essentially independent of immersion time. Thus substantially equivalent corrosion rates were obtained at the time of first immersion into the test sample corrosion liquor (the first point shown) as was achieved later. The essentially constant corrosion rate displayed by the pre-corrosion treated samples is considered to reflect the fact that owing to the pre-corrosion treatment stable grain dropping occurred from the outset in such cases. Grain dropping was confirmed by the observation of fine soot-like particles in the bottom of the test vessels at the end of the first and all subsequent test periods. The variation in corrosion rate of each pre-corroded specimen throughout the test period is believed to be due to chance fluctuations in the number of grains dropped from the samples during each period.

Similar results to these have been achieved from other test liquors illustrating that the pre-treatment quickly provides samples which then exhibit steady state corrosion rates of the appropriate level to allow accurate prediction of corrosion in plant in practice. Use of a simple pre-corrosion treatment prior to the actual corrosion testing ensures the establishment of stable grain dropping, and hence considerably shortens the test time required to obtain useful gravimetric data. The necessary accuracy, with greatly reduced testing times results.


### Sample Preparation and Corrosion – Investigations into Anisotropic Corrosion

To investigate the effects of anisotropic corrosion a series of 18/13/1 stainless steel specimens of the described in the initial preparation of samples section were further prepared to the design illustrated graphically in row A of Figure 6. The reference to RD in

row A is to the rolling direction of the steel during its initial manufacture, a direction discernable from visual inspection of a sample.

By way of illustration Figure 7 illustrates the concept of rolling direction, RD, thickness, T, and width, W, arising from the rolling operations involved in the production of steel which is then subsequently formed into pipes, process vessels or other structures.

Specimens 2 to 7 were ground to 800 grit and then pre-corroded in boiling 8M $HNO_3$ containing 1g/l Cr(VI) as detailed above. In order to determine the corrosion rate of the desired surfaces only, all of the surfaces of the pre-corroded specimen were then wet ground to 800 grit using SiC paper, except for the shaded surfaces in Figure 6. Some of the surfaces were formed by cutting and then grinding. In such cases approximately 1cm was removed from the end of each sample so as to eliminate any end grain corrosion penetration.

The row B samples of Figure 6 all had nominal dimensions of 15x10x4mm. Digital callipers were used to measure the exact dimensions of the sample and a test sample, sample 1 in row B, for which all samples have been ground to 800 grit was also deployed.

Sample 2 was used to determine the corrosion rate of an entirely pre-corroded specimen, with corrosion rates of the end, side and plate surfaces and of a 1.5mm diameter hole being determined from specimen 3 to 6. Specimen 7 was used to test the corrosion rate of the plate surface as the drilling of the hole through specimen 5 may have given to a higher corrosion rate due to residual burs or the like.

The samples were cleaned in methanol and weighed prior to immersion in the corrosion test liquor, which in this case consisted of 8M nitric acid and the other components provided above, at solution temperatures of 80°C and 100°C.

After 24 hours the specimens were removed, washed in de-ionised water and then methanol, dried and re-weighed. The corrosion test carried out at 80°C was then continued for a further 96 hours in order to obtain larger and hence more accurate weight losses.

## Test Results - Investigations into Anisotropic Corrosion

Based on the weight loss per surface area of the ground specimen, sample 1, it was possible to de-convolute the corrosion rate of the pre-corroded areas in specimens 3 to 7. The weight loss originating from the pre-corroded area of each specimen was calculated

by subtracting the calculated weight loss attributed to the ground surface from the experimentally measured weight loss. The corrosion rate of the pre-corroded surface was then calculated from this weight loss and the nominal area of the pre-corroded surface.

The results obtained, after subtracting the small amounts attributed to the ground surfaces, are represented for each face in Table 2. This data shows that the corrosion rate of the various faces increases in the order of plate, side and end (as schematically marked in Figure 7).

Table 2 also shows the predicted average corrosion rate of an entirely pre-corroded specimen calculated from the measured corrosion rates of the individual pre-corroded faces in the nominal surface areas. This is then compared with the actual average rate measured at the two temperatures. The discrepancy at 100° is believed to arise from weighing errors, which are very sensitive contributors to the measurements.

Table 2

|  | Area (cm$^2$) | 80°C | | 100°C | |
|---|---|---|---|---|---|
|  |  | Rate (mm/yr) | Predicted | Rate (mm/yr) | Predicted |
| Plate | 2.965 | 0.095 |  | 0.638 |  |
| Side | 1.200 | 0.134 |  | 0.916 |  |
| End | 0.800 | 0.258 |  | 1.277 |  |
| Hole | 0.188 | 0.145 |  | 0.924 |  |
| Whole | 5.153 | 0.129 | ·0.131 | 0.914 | 0.812 |

Table 3 provides an indication of the corrosion rate of each face expressed as a percentage of the average rate obtained from the measured weight loss of the entirely pre-corroded specimen normalised with respect to its entire nominal area.

Table 3

|  | 80°C | 100°C |
|---|---|---|
| Plate | 74 | 70 |
| Side | 104 | 100 |
| End | 200 | 140 |

| Hole | 112 | 101 |
|------|-----|-----|

At each temperature good correspondence between the plate and side rates individually compared with the overall rate were observed. This suggests insensitivity to corrosion conditions.

Based on this information it has been demonstrated that the corrosion rate into the plate surface can be reliably predicted from weight loss of the specimen containing exposed side and end grain surfaces also. To achieve this the time over which the test is performed is less than the time required for corrosion of the other surfaces in the test to switch from non-constant corrosion to a constant rate of corrosion. Indeed it is preferred that the time being as short as possible whilst achieving measurable weight loss. Whilst the factor may vary in its value to the particular grade of stainless steel tested and/or specimen dimensions, the position can be determined in each case. In particular it is suggested that for an 18/13/1 specimen, manufactured from the same cast as the specimens used in this set of experiments, but with dimensions RD x W x T, then the factor P, required to calculate the plate penetration rate from the gravimetric corrosion rate obtained from the testing of the whole (pre-corroded) specimen is given by

$$P = \frac{0.095(RD.W + RD.T + W.T)}{(0.095RD.W) + (0.134RD.T) + (0.258.W.T).}$$

Equivalent expressions can be formulated for the side grain and end grain relationships if desired.

**CLAIMS:**

1.      A method of corrosion testing metals, the method including providing a sample of the metal, the metal including grains, and contacting the sample of the metal with a test liquid, the rate of corrosion of the sample of metal in the test liquid being measured, wherein the sample of the metal is contacted with a treatment liquid prior to contact with the test liquid, the contact with the treatment liquid giving grain dropping from the metal.

2.      . A method according to claim 1 in which the treatment liquid includes nitric acid.

3.      A method according to claim 2 in which the nitric acid is of 5M or greater.

4.      A method according to any of claims 1 to 3 in which the treatment liquid includes one or more oxidants.

5.      A method according to claim 4 in which at least 0.5g/l Cr(IV) is provided in the treatment liquid.

6.      A method according to any pf claims 1 to 5 in which the sample is provided in contact with the treatment liquid for at least 12 hours.

7.      A method according to any preceding claim in which the sample is in contact with the treatment liquid until grain dropping is observed.

8.      A method according to claim 7 in which grain dropping is observed by visual inspection of grains below the sample in the treatment liquid and/or by a measured reduction in the size of the sample or one or more surfaces thereof and/or by optical examination of the sample.

9.      A method according to any preceding claim in which the test liquid includes nitric acid, the test liquid includes one or more dissolved species and causes intergranular corrosion of the metal to occur.

10.     A method according to any preceding claim in which the test liquid is provided at a temperature of at least 80°C.

11.     A method of corrosion testing metals, the method including providing a sample of the metal, the metal including grains, and contacting the sample of the metal with a test liquid, the rate of corrosion of the sample of metal in the test liquid being measured, wherein the sample of the metal is contacted with a treatment liquid prior to contact with the test liquid, the contact with the treatment liquid being for at least 6 hours and the treatment liquid including at least 5M acid and one or more oxidants.

12.     A method of establishing the rate of corrosion for a first surface of multi-surfaced metal in contact with a test liquid, the method including contacting a first test sample of the metal with the test liquid, the first test sample having a first surface which experiences constant rate corrosion on initial contact with the test liquid and other surfaces which undergo non-constant corrosion on initial contact with the test liquid and contacting a second test sample of the metal with the test liquid, the second test sample being of equivalent form to the first test sample, the second test sample having all surfaces which undergo non-constant corrosion on initial contact with the test liquid, the first and second test samples being contacted with the test liquid for the same time period, the differences in metal loss between the first sample and the second sample being attributed to the first surface of the first sample.

13.     A method according to claim 12 in which the rate of corrosion of the first surface is obtained by attributing the metal loss of the second sample evenly to all surfaces to provide a metal loss per unit area value and applying the same metal loss per unit area to the non-first surfaces of the first sample to give a non-first surface metal loss, the remaining metal loss from the first sample being attributed to the first surface of the first sample.

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14.    A method according to claim 12 or claim 13 in which the first surface is an external surface formed during production of the metal, particularly the surface contacted by the rollers.

15.    A method according to any of claims 12 to 14 in which the first surface experiences constant rate corrosion on initial contact due to previous contact with a treatment liquid.

16.    A method according to any of claims 12 to 15 in which the other surfaces experience non-constant corrosion on initial contact due to the surfaces not having contacted the treatment liquid, or more preferably due to the removal from that surface of the part of the surface which has been corroded by contact with the treatment liquid.

17.    A method according to any of claims 12 to 16 in which the time period is less than the time period for the corrosion of the other surfaces to change from the non-constant rate of corrosion to a constant rate of corrosion.

18.    A method according to any of claims 12 to 17 in which the method is applied to each of the situations where the plate, side and end surface is the first surface, the results for the three surface types are used to calculate an overall thickness loss or corrosion rate for the sample and this is compared with the result from a test sample in which all surfaces are provided so as to corrode at a constant rate from initial contact with the test liquid.

19.    A method according to any of claims 12 to 18 in which a ratio of the corrosion rate of the plate surface to the end surface and/or the side surface may be obtained.

20.    A method according to any of claims 12 to 19 in which a ratio of the rate of corrosion of the plate surface to the rate of corrosion of the overall sample is obtained.

21.    A method according to any of claims 12 to 20 in which the results are verified by conducting the method at different temperatures and/or different strengths of acid and/or different dissolved species and/or different oxidants in the test liquid.
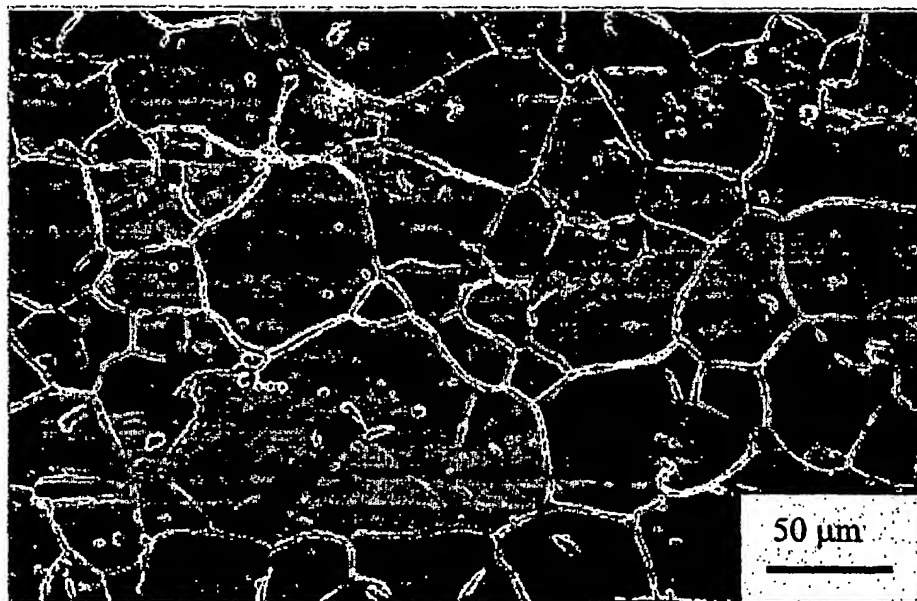
Scanning electron micrograph showing light etching of grain boundaries.
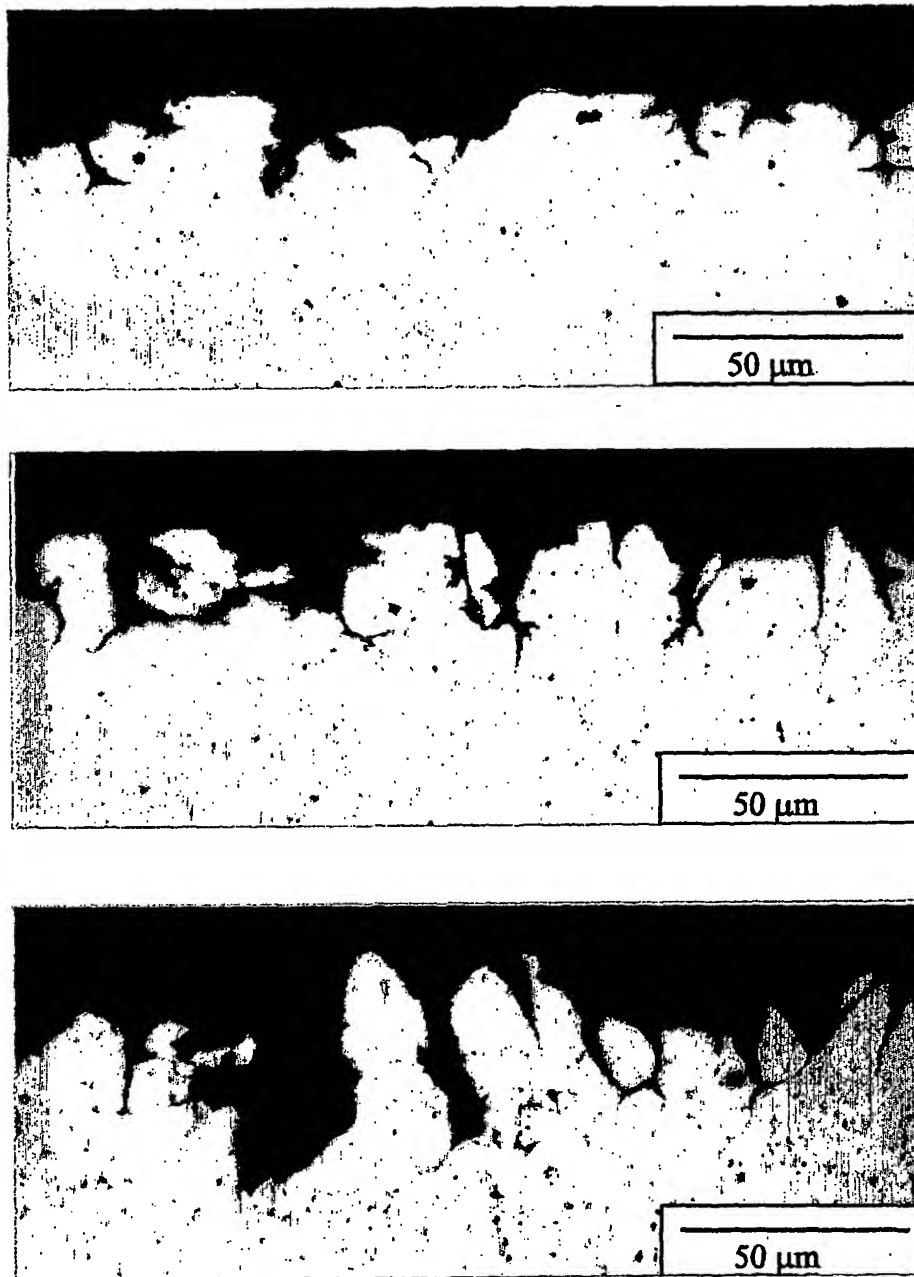
FIG.    1



Scanning electron micrograph showing the morphology of attack after
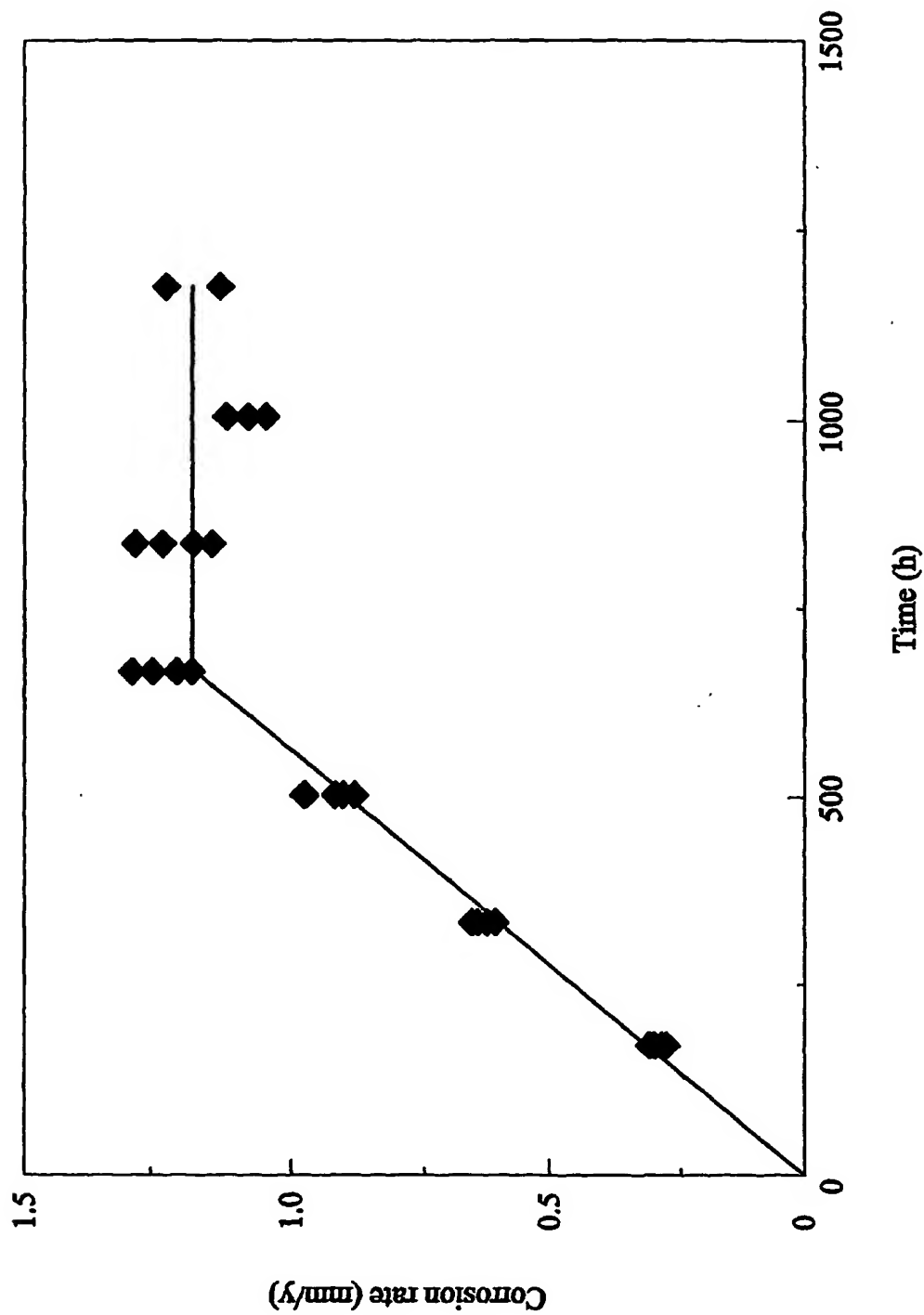the onset of grain dropping.

FIG.    2

Dissolution of (a) the plate surface, (b) the cut surface parallel to the
rolling direction and (c) the cut surface perpendicular to the rolling
direction of 18/13/1 stainless steel after corrosion testing in nitric acid
solutions.

## FIG.   3

FIG. 4

FIG. 5

FIG. 6



FIG. 7